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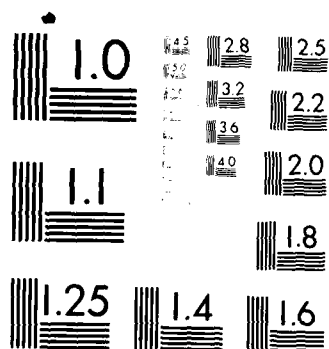
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TECHNICAL REPORT NO. 4

A Measurement of Intrinsic  $\text{SiO}_2$  Film Stress Resulting  
from Low Temperature Thermal Oxidation of Si

by

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in

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# ABSTRACT

A parallel beam reflection technique has been developed in our laboratory for measuring intrinsic residual stress for Si wafers thermally oxidized at temperatures of 600-1150°C. A detailed description of this technique is provided, and calculation of stress values are consistent with those reported in the literature. Low temperature thermal oxidations resulted in compressive intrinsic SiO<sub>2</sub> stresses greater than  $4 \times 10^9$  dyn/cm<sup>2</sup>.

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## INTRODUCTION

Over the past several years there have been a number of studies that have reported an intrinsic  $\text{SiO}_2$  film stress resulting from the thermal oxidation of Si at low oxidation temperatures (1,2,3). The intrinsic stress has been considered to be influential in Si oxidation kinetics both at the Si- $\text{SiO}_2$  interface (4,5) and by altering the diffusion of oxidant (6,7,8). Various stress measurements have been reported using light in single beam (9,10) and double beam (1,2) reflection experiments, and using the automatic Bragg angle camera (ABAC) technique (11,12). The descriptions of the ABAC technique are rather detailed, and although requiring relatively complex equipment, seem very adequate in measuring the strains (5). However, the simpler laser techniques are not as well described in the literature, often without calibration standards, diagrams and detailed procedures. We report, in the present study, a double beam technique, with calibration standards and the experimental results of one study using the technique to measure  $\text{SiO}_2$  stress on Si at room temperature. This study confirms previous measurements (1-3) and provides a simple reliable method to measure wafer strains resulting from film stress or other deformations.

## EXPERIMENTAL PROCEDURES

### Wafer preparation and film growth.

Strain measurements were performed on commercially obtained p-type (100)-oriented Si wafers. The RCA method (13) was used to clean the samples prior to oxidation, and the wafers were oxidized in dry  $O_2$  at temperatures ranging from 600-1150°C under atmospheric and high pressure conditions. Oxides were grown in the range of approximately 500-10,000 Å and their thickness was measured ellipsometrically. After oxidation, the oxide was stripped from the unpolished side of the wafer by waxing the front side and etching in a concentrated HF solution. Radius of curvature measurements as described below were recorded on these samples, then the remaining oxide was removed and the bare samples were remeasured to account for any warpage initially present in the Si substrate.

### Strain measurement apparatus and procedures.

We report a visible light reflection technique that was developed in this research to study film stress as a function of oxide thickness and oxidation temperature. The strain was determined by measuring the curvature change resulting from the thermal oxidation of Si wafers. Growth of an  $SiO_2$  film on a Si substrate bends the wafer such that the polished film-covered side is convex, thus denoting a compressive film stress (negative by convention). The parallel beam configuration shown in Fig. 1 is used to measure the radius of curvature of the samples. A plate-type beamsplitter,  $BS_1$ , is used to split the light beam from a He-Ne research laser (632.8 nm) into two separate beams. The resulting beams are 90° apart, and are reflected from laboratory quality

mirrors (1-3  $\lambda$  surface flatness) onto an Al-coated right angle prism. By alignment of the mirrors ( $M_1$  and  $M_2$ ), the resulting parallel beams are made to strike an oxidized Si wafer that is mounted on a translation stage, then the beams are reflected back to a second beamsplitter,  $BS_2$ . This beamsplitter allows both transmission and reflection of light, and thus prior to the parallel beams striking the sample, the beams are reflected to the screen and serve as a reference for aligning the parallelism of the beams. The rays are transmitted through  $BS_2$  and are reflected by the sample and then by  $BS_2$ , and are directed to another larger flat ( $5\lambda/\text{in.}$ ) mirror,  $M_3$ , located several meters from the sample, then directed back to the screen where their separation is measured and compared to the reference. If the oxidation process results in warpage of the wafers then a deviation in the initial parallel separation of the laser beams is observed.

The radius of curvature  $R$  of the sample is calculated from the equation

$$R = 2Lx/\delta$$

where  $x$  is the original ray separation (1.5 cm) for 1" wafers,  $L$  is the total path length (8 m) traveled by the reflected laser beams from the sample to  $M_3$  and to the measurement screen, and  $\delta$  is the deviation of the laser beams from their original separation. The radii of curvature for the  $\text{SiO}_2$  covered Si wafers actually measured were in the range of about 15 to 50 m normally. Therefore, for calibration, two standard mirrors with known radii of curvature



( $20 \pm 1$ ,  $40 \pm 2$  m) were used to test the accuracy and precision of this method and both were found to be in agreement (<5% difference) throughout the experiments. An optical flat was used in place of the sample to check the parallelism of the rays during the experiments.

#### EXPERIMENTAL RESULTS AND DISCUSSION

Once the strain in the sample is determined using the method reported above, the stress can be obtained from a simple Hooke's law relationship, where stress and strain are proportional through Young's modulus of elasticity. This implies elastic deformations only and that the resulting stress distribution is isotropic. The average compressive film stress can be derived from Stoney's equation (14) as

$$\sigma_f = Et_s^2 / (1-\nu)t_f R$$

where  $t_s$  and  $t_f$  are the thickness of the substrate and oxide, respectively, and  $E/(1-\nu)$  is taken as  $1.805 \times 10^{12}$  dyn/cm<sup>2</sup> for (100) Si (15), where  $E$  and  $\nu$  are Young's modulus and Poisson's ratio, respectively. Since the measurement is taken at room temperature, the total film stress is the sum of the thermal-expansion and intrinsic stress. The thermal-expansion stress, which results from a difference in the thermal expansion coefficients between Si and SiO<sub>2</sub>, is zero at the oxidation temperature and develops as the sample cools. Thermal-

expansion stress values are calculated from the relationship:

$$\sigma_{th} = \frac{\Delta\alpha \Delta T E}{(1-\nu)}$$

where  $\Delta\alpha$  is the difference between the thermal expansion coefficients for Si (16) and  $\text{SiO}_2$  (17) obtained from the literature, and  $\Delta T$  is the difference between the oxidation temperature and room temperature.

The total film stress as a function of  $\text{SiO}_2$  thickness is shown in Fig. 2, as obtained from strain measurements on samples grown at temperatures ranging from 900 to 1100°C. Scatter in the measured values of stress is observed for thinner oxides, because the thinner oxide does not impart enough compressive force to bend the wafers sufficiently to allow accurate and reproducible measurements of R. For 8-11 mil. thick substrates used, it was usually found that at least 1000 Å was required to obtain sufficient curvature for accurate strain measurements. A constant stress results for films thicker than 1000 Å for samples oxidized above 1000°C, which is comparable to previously reported values (11,18-20).

As noted previously, the film stress is a sum of the thermal-expansion stress and intrinsic stress when measured at room temperature. Fig. 3 shows these two stress components as a function of oxidation temperature for (100) Si wafers. It is interesting to note that a large difference between the curves arises at lower oxidation temperatures and can be understood in accordance with a viscous flow model previously reported (3). In this model, the  $\text{SiO}_2$  film is

assumed to behave as a Maxwell viscoelastic solid. If the oxidation temperature is high enough, then the oxide viscosity is sufficiently low to allow  $\text{SiO}_2$  to flow freely in the direction normal to the wafer. For lower process temperatures ( $<950^\circ\text{C}$ ), the oxide viscosity is high, and no stress relief mechanism such as viscous flow is available. This leads to a buildup of intrinsic stress since the molar volume change in converting Si to  $\text{SiO}_2$  is constrained by adhesion to the plane of the wafer. Evidence for the existence of intrinsic stress has been reported at lower temperatures by EerNisse (2), and a viscous flow model has also been reported (1) which agrees with the data shown in Fig. 3. The intrinsic stress or difference between the two curves in Fig. 3 is plotted in Fig. 4 for wafers oxidized under atmospheric and high pressure conditions. This curve clearly shows an intrinsic stress buildup at lower oxidation temperatures.

#### SUMMARY AND CONCLUSIONS

We have reported the details of a parallel laser beam method to measure wafer deformations, including calibration and alignment procedures. This technique was used to obtain  $\text{SiO}_2$  stress on Si by the direct measurement of the Si substrate radius of curvature, i.e. the film strain after  $\text{SiO}_2$  growth. Our results are concordant with previous ABAC stress measurements, and the laser technique is shown to yield reliable and reproducible stress results. The intrinsic  $\text{SiO}_2$  film stress on (100) Si is observed to increase from zero for temperatures above  $1000^\circ\text{C}$  to greater than  $4 \times 10^9 \text{ dyn/cm}^2$  at  $600^\circ\text{C}$ , while the total room temperature film stress is about  $3 \times 10^9 \text{ dyn/cm}^2$  above  $1000^\circ\text{C}$ .

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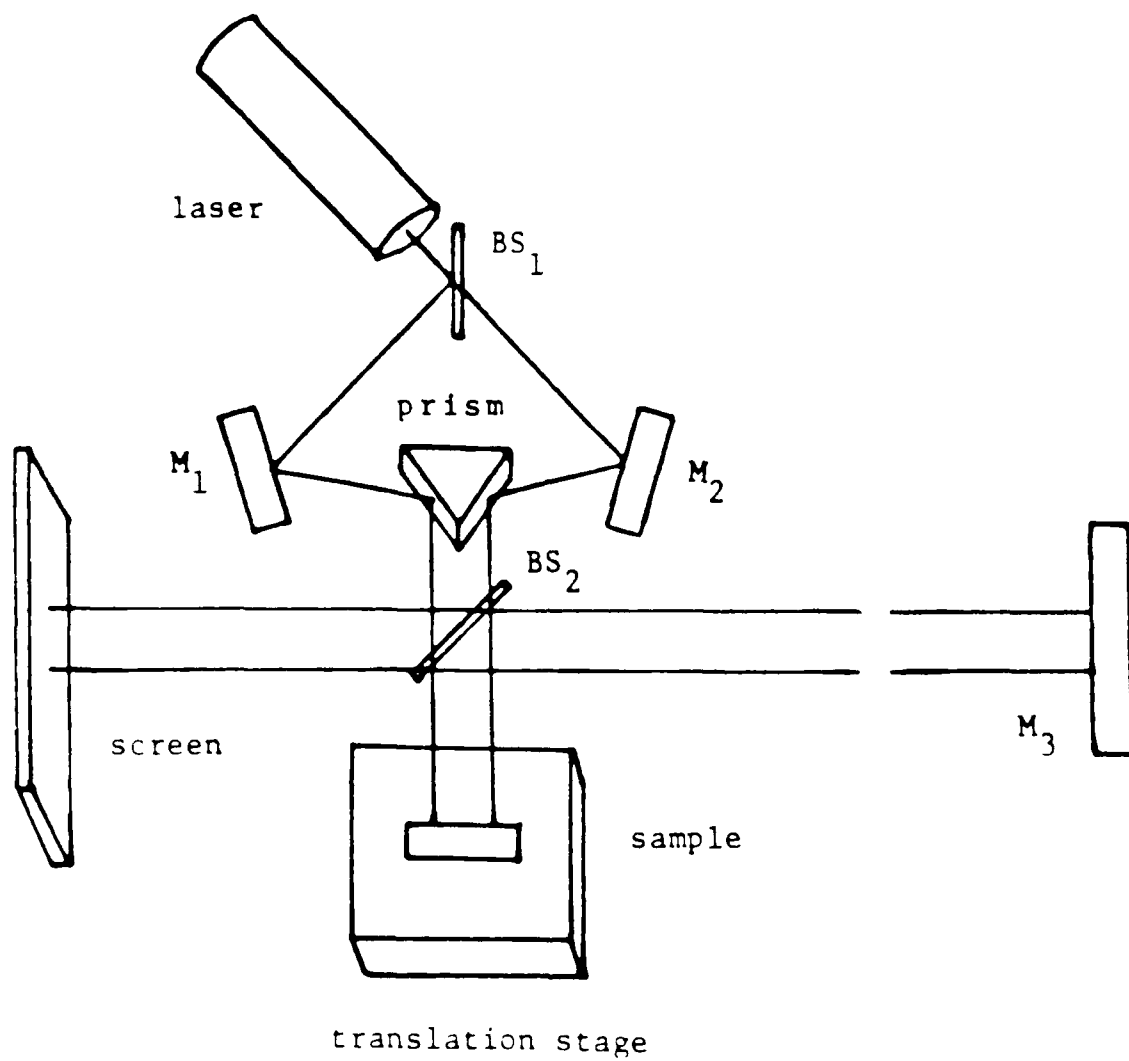
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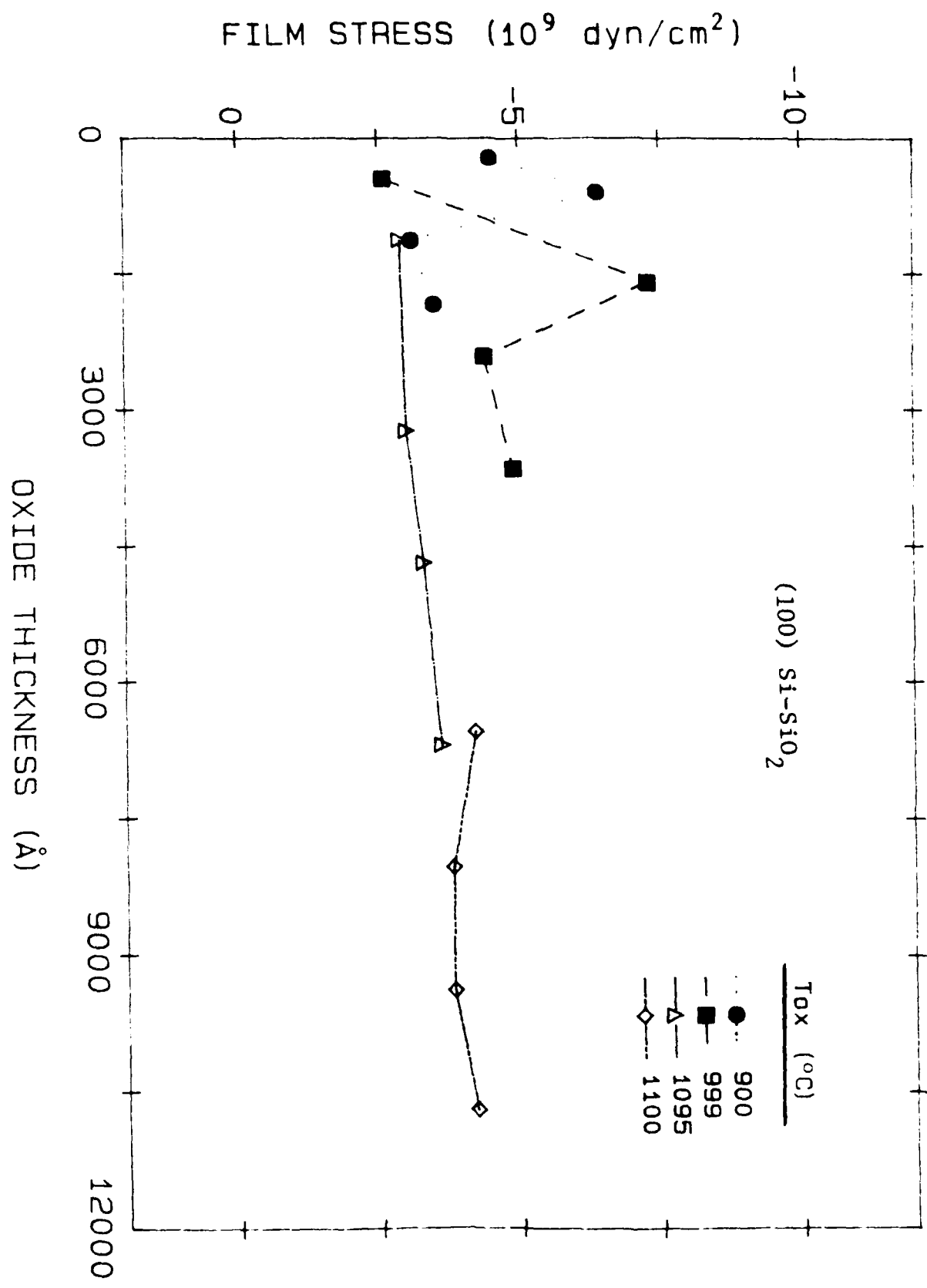
Figure 1. Parallel-beam configuration for measurement of wafer curvature.  
(BS = beamsplitter, M = flat mirror).

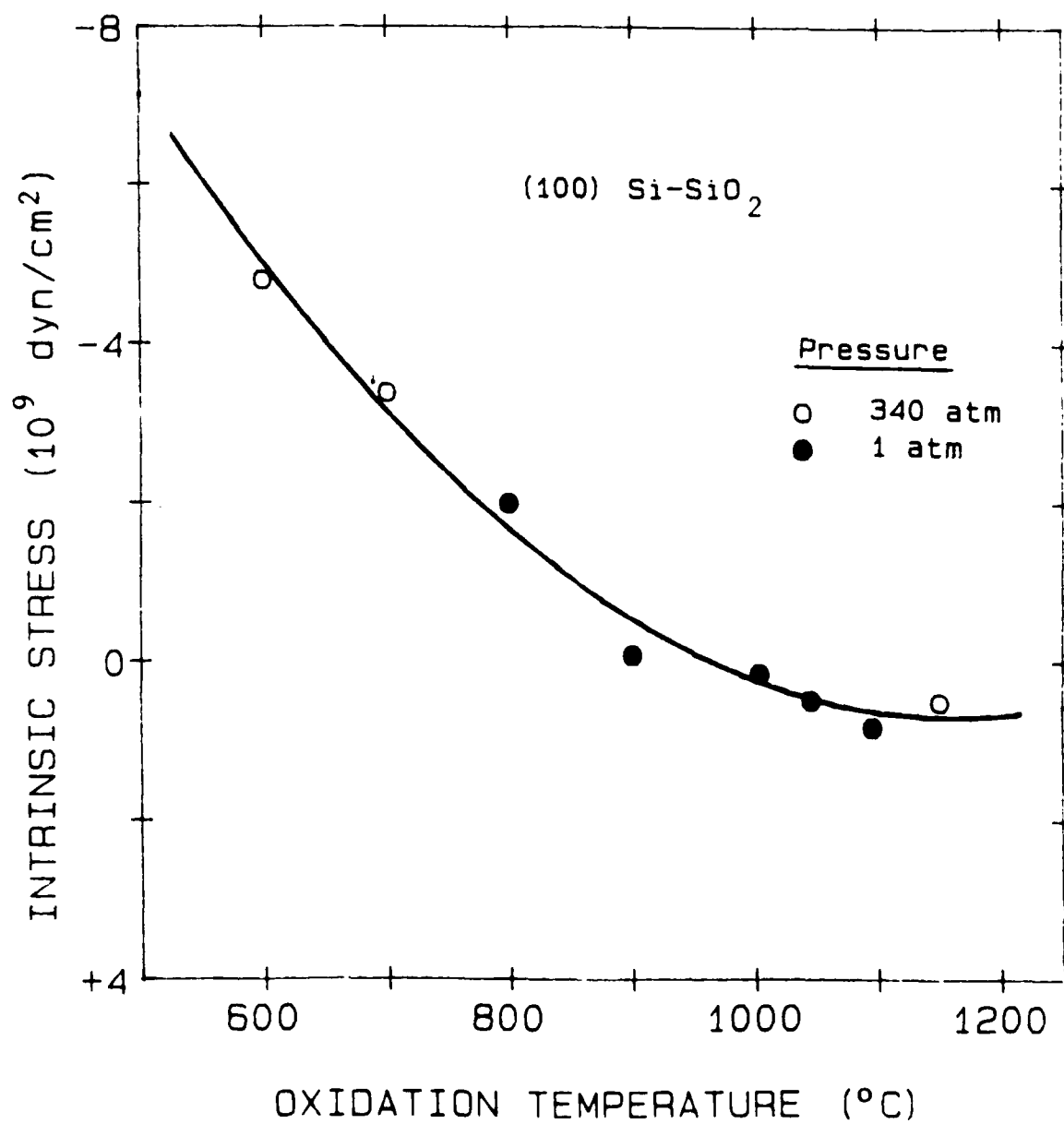
Figure 2. Total film stress measured as a function of oxide thickness.

Figure 3. Components of stress vs. oxidation temperature for (100)-oriented samples.

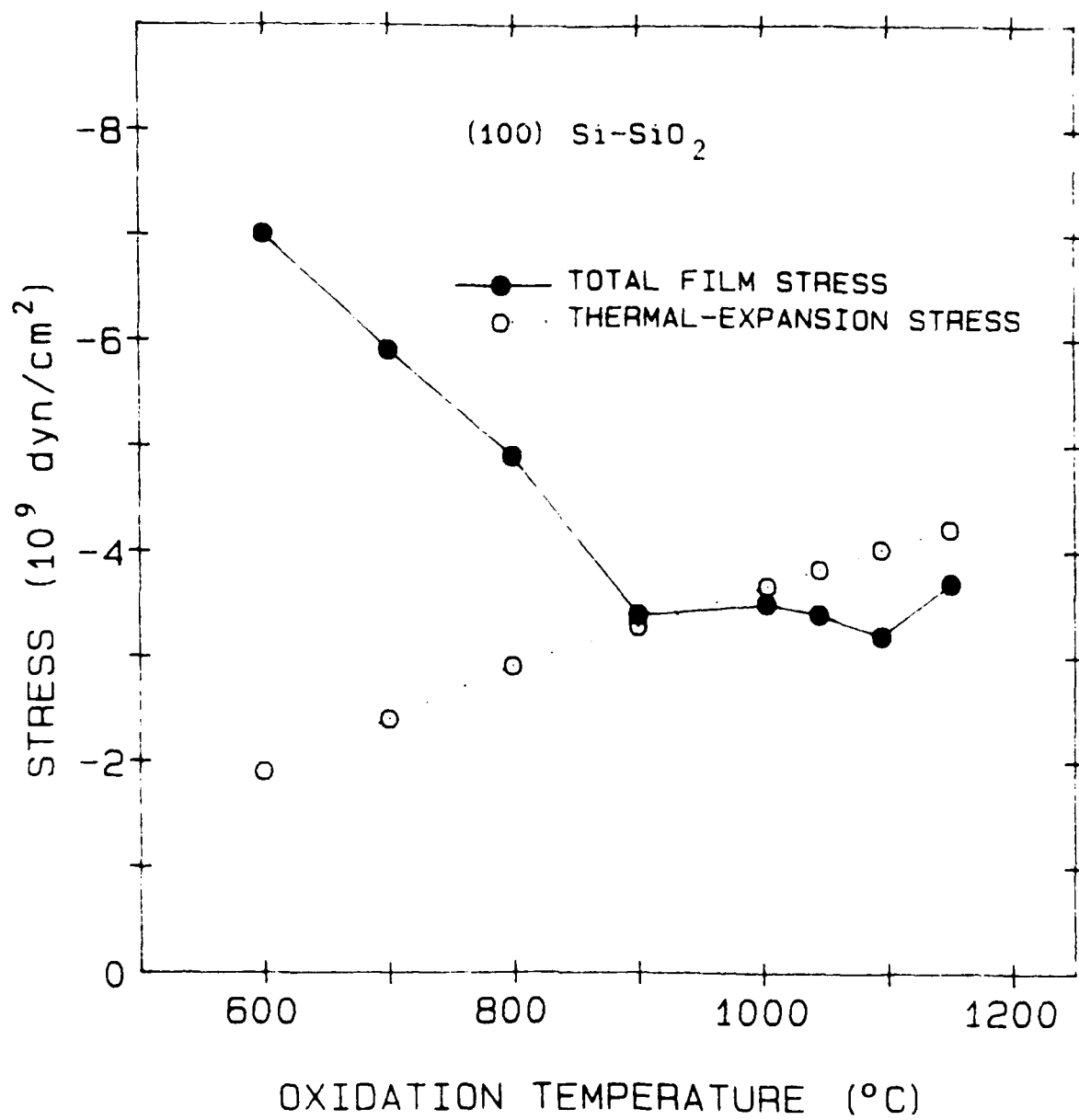
Figure 4. Intrinsic stress vs. oxidation temperature for (100)-oriented samples.











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